(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
4 October 2001 (04.10.2001)

PCT

(10) International Publication Number
WO 01/73338 A1

(51) International Patent Classification[7]: F16L 49/00, A61M 39/10, G02B 6/44, G01N 30/60, F16J 15/06

(21) International Application Number: PCT/AU00/01256

(22) International Filing Date: 16 October 2000 (16.10.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
PQ 6469 27 March 2000 (27.03.2000) AU

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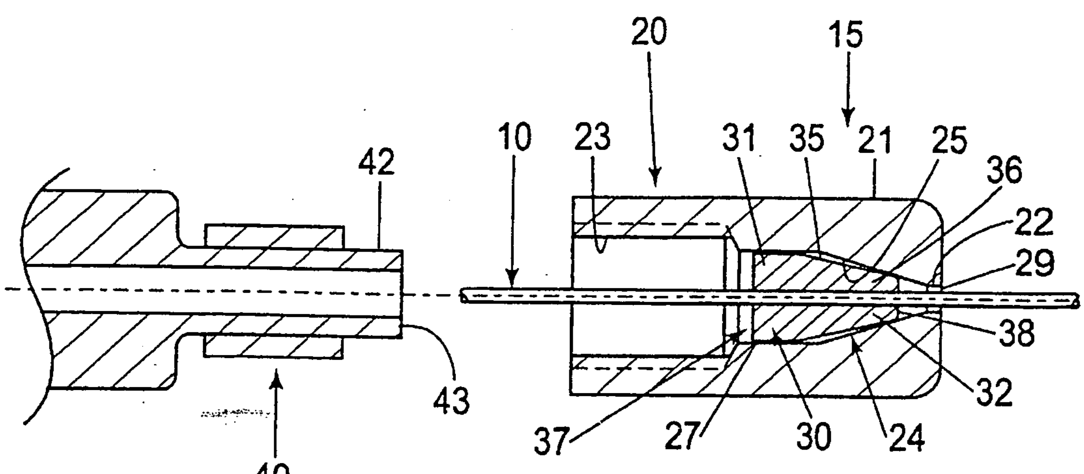
(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: FERRULE SEAL



(57) Abstract: Seal apparatus for sealing about a fracturable tube, includes a ferrule (30) with an axial bore (32) to receive a tube, and a longitudinally bored outer component (20) having a cavity (24) to co-axially receive the ferrule. The ferrule has an external surface (35) that tapers to a tip region (36) defining one axial end of the ferrule. The cavity has a peripheral wall (25) that tapers with a steeper taper angle than the tapered surface of the ferrule, to a cross section smaller than the tip of the ferrule. The cross-section of the tip region and the relative taper angles and hardnesses of the tapered surface and the tapered wall are selected whereby, on relatively drawing the tapered wall of the outer component cavity onto the tapered surface of the ferrule, the tip region is deformed for sealing gripping a protectively coated fracturable tube traversing the bore of the ferrule, without fracturing the tube.

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FERRULE SEAL

Field of the Invention

This invention relates generally to the sealing of fittings about fracturable tubes and as such is of particular, though not exclusive, interest in the sealing of fittings about fused silica capillary tubing in gas and liquid chromatography. The invention is also applicable to the sealing of fittings for optical fibres.

Background Art

The analytical techniques of gas and liquid chromatography and mass spectrometry are achieved with instruments which may be operated at elevated temperatures and relatively high pressure or even vacuum. Fused silica capillary tubing may be an integral component of such instruments, especially for providing separation columns. Commonly for gas chromatography, and in some circumstances for liquid chromatography, separation columns comprise fused silica capillary tubing. There are typically an inlet and an outlet end to this separation column. Fused silica tubing is also used for transfer tubing in a variety of analytical chemistry applications.

The sample to be analysed by gas chromatography is introduced into the inlet end of the separation column and carried through the column by a carrier gas which is necessarily of high purity. To retain the sample to be analysed in the gas phase, it is usually necessary to heat the column to an elevated temperature. The inlet end of the column is usually above atmospheric pressure to allow the gas to be forced through the column. Consequently, the connection at the inlet end of the column needs to be gastight so as not to allow leakage from the column or introduce contamination into the column. The outlet end of the column can be connected to a number of devices, usually a detection device of some sort, and can also be the inlet of another column. The outlet end of the column may be at, below or above atmospheric pressure. The connection at the outlet end of the column will also be gastight to avoid introduction of contamination or leakage.

To seal the column at both inlet and outlet ends, conventional fittings employ ferrules that deform onto the outside of the column to make a substantially leaktight seal with the column as well as with the fitting, thereby forming a seal against the outside atmosphere.

A sealing system of general application to fittings for tubing systems is the Swagelok tube fitting available from the Crawford Fitting Company. In this system, a threaded driving component is rotated to push a metal ferrule, with a conical tip, into a surrounding component with a cavity of matching taper angle. The ferrule thereby bites into the tube being sealed: if applied to a fracturable tube such as a fusica silica tube, this seal assembly will promptly break the tube.

Historically, when capillary columns were metal tubes, various types of ferrules were used including metal ferrules. Glass columns were then introduced and soft materials were required as the ferrule material. These soft materials initially included silicone rubber and teflon, and in some rare instances lead, and then subsequently graphite became the usual material. In 1979, fused silica capillary columns were introduced. These involved a very high purity form of glass with an exterior coating of a polymer, usually a polyimide or a thin metal to protect the fused silica from being damaged. Because the column material was still a glass that could easily be broken, ferrule materials that remained in use with fused silica columns were either graphite or Vespel (trade mark), or Vespel incorporating a significant percentage of graphite to make the ferrule softer. All of these types were designed to readily deform to the shape of the column tube.

Both graphite and the various types of vespel ferrules have recognised deficiencies. Graphite ferrules are permeable to oxygen which causes problems with detectors and damage to capillary columns. Furthermore graphite is an extremely absorptive material and will absorb sample constituents that are being analysed in the chromatographic system that it comes in contact with. The ferrule should not normally come in contact with sample components but it is recognised as a problem that pieces of graphite can become detached or extrude into parts of the chromatographic system thus destroying the integrity of the chromatographic separation.

Whenever a capillary column is inserted through a graphite or vespel ferrule to make a connection, a section of the column must be cut off in case there are particles of ferrule material around the end of the column.

The main problem with vespel and graphite-impregnated vespel ferrules is that after an initial leaktight seal is made and the chromatographic system is heated, as is required in chromatographic analysis, and then the system is cooled down in preparation for the next analysis, the vespel ferrule develops substantial leaks necessitating the retightening of the fitting to reestablish the leaktight seal. On subsequent temperature cycles a leak may or may not develop.

The thermal coefficients of expansion of vespel materials are significantly higher than those of the metal components encapsulating them. This puts the ferrule under significant compression causing it to creep dimensionally. As the fitting system is cooled the vespel ferrule contracts more than the metal fitting encapsulating it which causes gaps and leakage paths to be formed.

Attempts to address this problem are disclosed in US patents 4991883 and 5234235 and include spring-loaded self-compensating ferrules.

These various deficiencies of existing graphite and vespel ferrule systems have diminished the reliability of chromatography systems, leading to compromised performance as well as non-productive time in rectifying the faults. In addition, damage to components in the systems regularly occur when operators apply excessive force to fittings in efforts to eliminate leaks.

It is therefore an objective of the present invention to provide an improved seal assembly for sealing about a fracturable tube such as a tube of glass or fused silica that at least partially alleviates the aforementioned problems.

Summary of the Invention

The invention entails an appreciation that, by appropriate balancing of interrelated parameters of a ferrule and an outer component for deforming the

ferrule, and taking account of the coating typically found on fracturable tubes s[ ] as capillary column tubes, it is possible, in a preferred embodiment of the invention, to revert to a metal ferrule and to at least in part alleviate the problems encountered with existing graphite and vespel ferrule systems.

The invention accordingly provides seal apparatus for sealing about a fracturable tube, including a ferrule with an axial bore to receive a tube, and a longitudinally bored outer component having a cavity to co-axially receive the ferrule. The ferrule has an external surface that tapers to a tip region defining one axial end of the ferrule. The cavity has a peripheral wall that tapers with a steeper taper angle than the tapered surface of the ferrule, to a cross section smaller than the tip of the ferrule. The cross-section of the tip region and the relative taper angles and hardnesses of the tapered surface and the tapered wall are selected whereby, on relatively drawing the tapered wall of the outer component cavity onto the tapered surface of the ferrule, the tip region is deformed for sealing gripping a protectively coated fracturable tube traversing the bore of the ferrule, without fracturing the tube.

Preferably, the tapered surface of the ferrule is softer than the tapered wall of the outer component cavity.

Preferably, the materials of the outer component and ferrule are similar but of differing hardnesses to achieve the result just described. Preferred such materials are metal. A particularly advantageous material is stainless steel and it is preferred that the same grade of stainless steel be used. To achieve the preferred requirement that the ferrule tapered surface is softer than the wall of the surrounding cavity of the outer component, the ferrule may be subjected to a suitable pre-treatment, for example an annealing treatment.

The ferrule is preferably of annular cross-section, and its tapered surface therefore conical in form. The tapered wall is then an inverted cone to match.

The outer component preferably has an outer surface formation to facilitate tool engagement for rotation of the component. The outer component may

conveniently include a thread formation for engaging another threaded component, whereby relative rotation of the two can effect said drawing action.

Preferably, the tapered surface of the ferrule and the tapered wall of the outer component cavity are treated to minimise friction in their interengagement and thereby to also facilitate disengagement. Suitable treatments for this purpose include silverplating, or application of a coating of molybdenum disulphide. The coating may, for example, be of a thickness 0.5 to 5 micron, typically about 1 micron.

Preferably, said tip region is rounded at the end of the ferrule.

Advantageously, the ferrule further includes an annular sealing surface at its other axial end which lies in a diametral plain with respect to the axis of the system. The sealing of this end surface is preferably enhanced by an integral peripheral rib or other land or taper to contact an adjacent fitting and surface.

The invention further extends to a combination of an assembled seal apparatus as aforedescribed and a frangible tube traversing the ferrule bore, the frangible tube having a protective coating, eg. of thickness 5 to 100 microns.

Brief Description of the Drawings

The invention will now be further described, by way of example only, with reference to the accompanying drawings, in which:

- Figure 1 is an axial cross-section of seal apparatus as a seal assembly according to an embodiment of the invention, shown in position on a fused silica tube ready for co-operation with a spigot component of a fitting to be sealed by the assembly;

- Figure 2 is an enlargement of a portion of Figure 1 following activation of the seal assembly; and

- Figure 3 depicts cross-sections of alternative modified ferrules for the seal assembly of Figure 1, each having sealing formations on the rear angular surface of the ferrule.

Preferred Embodiments

The illustrated seal assembly 15 includes an outer component in the form of an elongated nut 20 with an axial bore 22 and a hexagonal section outer surface 21 to facilitate tool engagement. Bore 22 has a threaded counterbore 23 at one end and, extending between counterbore 23 and the other end, a co-axial cavity 24 in part defined by a tapered wall 25 of inverted conical form. The other principal component of seal assembly 15 is a ferrule 30 with an axial bore 32 and, extending over most of its length, an external surface 35 that tapers to a tip region 36 at one axial end face 38 of the ferrule. Ferrule 30 is of generally annular cross-section and so surface 35 is conical in form. The two components are preferably formed in stainless steel.

Cavity 25 is dimensioned to receive ferrule 30, while the bore 32 of the ferrule 30 is dimensioned to receive a typical fused silica glass tube 10 of the kind employed in gas or liquid chromatography. This tube may generally be of diameter 0.3 to 1.0mm outside diameter and typically has a protective coating 5 to 100 micron, eg. about 10 micron thick, eg. of metal, silicone or polyimide. The tube is typically a close but adjustable fit within the ferrule bore: for example a bore of 0.4mm for standard tube of outside diameter 0.32mm. The bore 22 of nut 20, which opens as a short aperture 29 from cavity 25 to the outer end of the nut, is somewhat larger in diameter than the tube (eg. twice as large) but smaller than the diameter than the tip 38 of the ferrule.

The threaded counterbore 23 of nut 20 provides a socket for a matching threaded spigot 42 of a union 40 with which the assembly is to be connected to form a fitting, eg an inlet or outlet fitting for a separation column provided by tube 10 in gas or liquid chromatography instrumentation.

Generally conical ferrule 30 includes a rear cylindrical portion 31 of larger

uniform diameter with a smoothly flat polished end face 37 defining the rear end of the ferrule, while the forward end of the ferrule is defined by end face 38 of tip region 36. Spigot 42 of union 40 defines an annular end face 43 which, in the fully assembled fitting, seals against rear end face 37 of ferrule 30. Cylindrical portion 31 is a close but not interference sliding fit with a uniform diameter rear portion 27 of cavity 24.

Three parameters of nut 20 and ferrule 30 are selected to achieve a satisfactory seal without fracturing, ie. either breaking or cracking, the tube 10. Firstly, the taper angle of tapered wall 25 of cavity 24 is substantially greater than the taper angle of conical surface 35, eg. about 1.2 to 1.5 times greater. Exemplary included angles for the respective surfaces would be 40-45° and about 30°. Secondly, although the materials of the ferrule and nut are selected to be the same grade of stainless steel, in order to minimise differences in thermal expansion coefficients, the material of ferrule 30 and therefore of conical surface 35 is arranged to be substantially softer than that of the nut and therefore of surface 25. This can be achieved, for example, by heat treating ferrule 30, and a suitable treatment is an annealing treatment to 1100°C. Thirdly, the diameter of the end face 38 of tip region 36 is selected to be sufficiently great to ensure the integrity of the tip region during deformation by the nut but sufficiently small to facilitate its deformation in the manner shortly to be described.

The seal assembly 15 is applied to a tube 10 traversing the ferrule as illustrated in Figure 1 by first applying a spanner or other tool to the nut to threadingly engage socket 22 with spigot 42. The nut is rotated until spigot end face 43 engages the rear face 37 of the ferrule and the tapered wall 25 of cavity space is relatively drawn onto the smaller end of conical surface 35 at tip region 36. The aforesaid parameters are selected so that, on further rotation of nut 20, the tip region 36 is deformed to sealingly grip the protective coating of tube 10 without fracturing the tube. The protective coating on tube 10 provides a layer into which end region 36 of the ferrule may bite and deform without adversely contacting the surface of the underlying glass material.

It is preferable that during the deformation stage friction between the components is minimal so that the cavity surface of the nut is able to slide over the conical ferrule surface as the tip region deforms. This is facilitated, as is disengagement of the components to undo the fitting, by precoating surfaces 25 and 35 eg. by silverplating or with a coating of a smooth-forming anti-friction medium such as molybdenum disulphide. Such a coating may be as little as 1 micron thick. It is also preferable for the circular edge at the front end of tapered wall 25 to be rounded in cross-section.

Figure 2 provides a magnified indication of the outcome of the tightening process. The frustoconical zone 50 of direct contact between the tapered surfaces is a relatively small proportion of the overall length of the surfaces in the axial direction, and the sealing zone 52 on the glass tube may be even smaller in axial extent.

It will be appreciated that an additional sealing region for the assembly is the flat rear surface face 37 in contact with the end face 43 of the spigot 42. Exemplary enhancements for improving the sealing between these surfaces are shown in Figure 3, ie an annular rib of semi-circular (A) or triangular (B) cross-section. Instead of a transverse flat end face, sealing may be effected with a conical interface so that the female is tapered or conical at both ends.

It will be understood that an excessive further rotation of nut 20 will eventually apply stress to the glass of the tube itself and cause it to fracture. By appropriate selection of the aforementioned parameters, the arrangement may be that a large radial turn of the nut can be tolerated from the point the ferrule has made a reliable seal onto the glass tube protective coating through to the point the glass fails due to sufficient distortion of the ferrule so that it is encroaching on the glass under the protective coating. By ensuring that this excessive tightening is substantial, eg of the order of 180° of turn, one can minimise the risk of accidental damage by users through overtightening of the fitting.

In a modified embodiment (not illustrated), the cavity 24 and its tapered wall are provided in spigot 42 of the union, and sealing end face 43 in nut 20 at the

Claims

1 Seal apparatus for sealing about a fracturable tube, including:

a ferrule with an axial bore to receive a tube, which ferrule has an external surface that tapers to a tip region defining one axial end of the ferrule; and

a longitudinally bored outer component having a cavity to co-axially receive said ferrule, said cavity having a peripheral wall that tapers with a steeper taper angle than said tapered surface of the ferrule, to a cross section smaller than said tip of the ferrule;

wherein the cross-section of said tip region and the relative taper angles and hardnesses of said tapered surface and said tapered wall are selected whereby, on relatively drawing the tapered wall of the outer component cavity onto the tapered surface of the ferrule, the said tip region is deformed for sealing gripping a protectively coated fracturable tube traversing the bore of the ferrule, without fracturing the tube.

2 Seal apparatus according to claim 1, wherein the tapered surface of the ferrule is softer than the tapered wall of the outer component cavity.

3 Seal apparatus according to claim 1 or 2, wherein the materials of the outer component and ferrule are similar but of differing hardnesses.

4 Seal apparatus according to claim 1, 2 or 3 wherein the materials of the outer component and ferrule are metal.

5 Seal apparatus according to claim 4, wherein said metal is stainless steel.

6 Seal apparatus according to any preceding claim, wherein the ferrule is subjected to a suitable pre-treatment, for example an annealing treatment, to make said tapered surface softer than the tapered wall of said cavity.

7 Seal apparatus according to any preceding claim, wherein the ferrule is of

annular cross-section, and its tapered surface therefore conical in form.

8 Seal apparatus according to any preceding claim, wherein the outer component has an outer surface formation to facilitate tool engagement for rotation of the component.

9 Seal apparatus according to any preceding claim, wherein the tapered surface of the ferrule and/or the tapered wall of the outer component cavity are treated to minimise friction in their interengagement and thereby to also facilitate disengagement.

10 Seal apparatus according to claim 9, wherein said treatment is silverplating, or application of a coating of molybdenum disulphide.

11 Seal apparatus according to any preceding claim, wherein the tip region is rounded at the end of the ferrule.

12 Seal apparatus according to any preceding claim, wherein the ferrule further includes an annular sealing surface at its other axial end which lies in a diametral plain with respect to the axis of the system.

13 Seal apparatus according to any preceding claim, in combination with a frangible tube traversing the ferrule bore, the frangible tube having a protective coating.

14 The combination of claim 13 wherein said protective coating is of thickness in the range 5 to 100 microns.

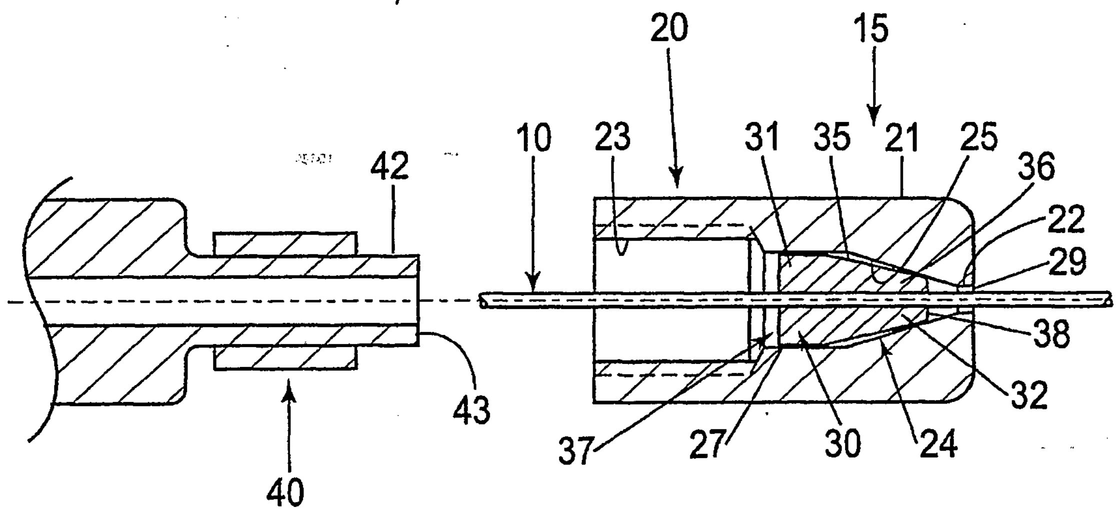
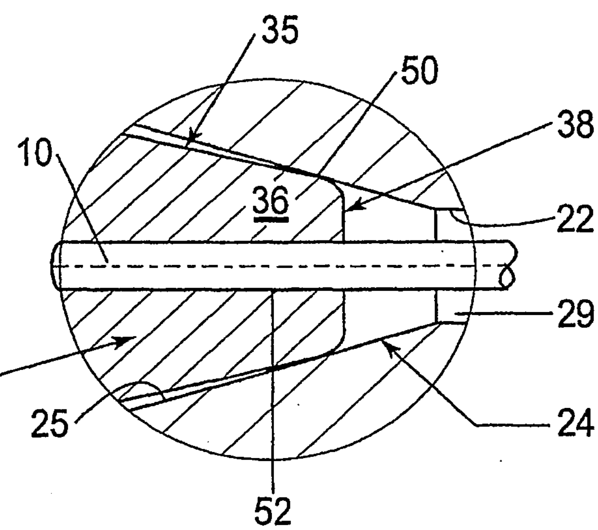


FIGURE 1

FIGURE 2



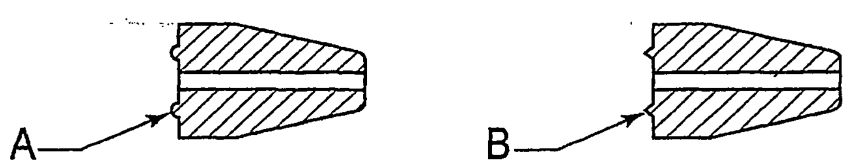


FIGURE 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU 00/01256

A. CLASSIFICATION OF SUBJECT MATTER

Int Cl[7]: F16L 49/00, A61M 39/10, G02B 6/44, G01N 30/60, F16J 15/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC WHOLE DATABASE

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
AU: IPC as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
DWPI and JAPIO with keywords: Pipe, tube, conduit, capillary, fibre; ferrule, connect:, fitting.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 288 113 A (SILVIS et al) 22 February 1994	
A	US 4 991 883 A (WORDEN) 12 February 1991	
A	US 4 876 005 A (AMERICA) 24 October 1989	

[X] Further documents are listed in the continuation of Box C

[X] See patent family annex

* Special categories of cited documents:

- "A" Document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier application or patent but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed
- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
23 November 2000	1 - DEC 2000

Name and mailing address of the ISA/AU	Authorized officer
AUSTRALIAN PATENT OFFICE PO BOX 200 WODEN ACT 2606 AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No.: (02) 6285 3929	RAJEEV DESHMUKH Telephone No.: (02) 6283 2154

INTERNATIONAL SEARCH REPORT

ıonal application No.
PCT/AU 00/01256

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4 529 230 A (FATULA, JR) 16 July 1985	

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU 00/01256

Information on patent family members

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
US	5288113	CA	2110537	EP	604018	JP	7306191
US	4991883	GB	2236369				
US	4876005	DE	68924284	EP	328146		
US	4529230	EP	87598				

END OF ANNEX